FINAL

WORK PLAN

with

QUALITY ASSURANCE PROJECT PLAN

for

SMALLMOUTH BASS ACOUSTIC TELEMETRY AND TISSUE SAMPLING FOR SPRING 2022

at

River Operable Unit, Bradford Island CASCADE LOCKS, OREGON

Prepared by

U.S. ARMY CORPS OF ENGINEERS
Portland and Seattle Districts



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TITLE AND APPROVAL SHEET WORK PLAN WITH QUALITY ASSURANCE PROJECT PLAN (WP-QAPP) SMALLMOUTH BASS ACOUSTIC TELEMETRY AND TISSUE SAMPLING FOR SPRING 2022

RIVER OPERABLE UNIT, BRADFORD ISLAND, CASCADE LOCKS, OREGON

This Work Plan with Quality Assurance Project Plan (WP-QAPP) describes sampling activities and Data Quality Objectives (DQOs) for smallmouth bass sampling and tracking at the River Operable Unit, Bradford Island, Cascade Locks, OR. The QAPP is based on the *Intergovernmental Data Quality Task Force Uniform Federal Policy for Quality Assurance Project Plans Guidance (EPA 2009)*.

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Kristen Kerns, Sampling Technic	cal Lead, NWS	Date
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Alison M. Suess, Ph.D., Chemis	t, NWS	Date

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TABLE OF CONTENTS

1.	PROJE	CT MANAGEMENT AND OBJECTIVES	9
1.1	l. Pro	oject Organization, Responsibilities and Authority	9
	1.1.1.	Communication Pathways	10
	1.1.2.	USACE Personnel Responsibilities and Qualifications	11
	1.1.3.	External Technical Review Personnel Responsibilities and Qualifications	13
1.2	2. Pro	oject Planning	13
	1.2.1.	Project Planning (Scoping)	13
	1.2.2.	Problem Definition, Site History, and Background	13
1.3	3. Pro	oject Quality Objectives and Measurement Performance Criteria	14
	1.3.1.	Development of Project Quality Objectives Using the Systematic Planning Process	14
	1.3.2.	Measurement Performance Criteria	20
1.4	I. Sec	condary Data Evaluation	20
1.5	5. Pro	ect Overview and Schedule	20
2.	DATA	GENERATION AND ACQUISITION	22
2.1	l. Sa	npling Tasks	22
	2.1.1.	Sampling Process Design and Rationale	22
	2.1.2.	Sample Collection Procedures	23
	2.1.3.	Sample Naming Convention	26
	2.1.4.	Decontamination Procedures	27
	2.1.5.	Field Equipment Calibration, Maintenance, Testing and Inspection Procedures	27
	2.1.6.	Supply Inspection and Acceptance Procedures	27
	2.1.7.	Field Documentation Procedures	27
	2.1.8.	Sample Delivery	28
	2.1.9.	Sample Custody	29
2.2	2. An	alytical Tasks	29
	2.2.1.	Analytical Methods	29
	2.2.2.	Analytical Instrument Calibration Procedures	29
	2.2.3.	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedure	es29
2.3	3. Qu	ality Control Samples	29
	2.3.1.	Field Quality Control Samples	29
	2.3.1.1	Field Duplicates	29

	2.3.1.2	. Trip Blanks	30
	2.3.1.3	. Equipment Rinse Blanks	30
	2.3.2.	Analytical Method Quality Control Samples	30
	2.3.2.1	. Method Blanks	30
	2.3.2.2	. Laboratory Control Samples (LCS)	30
	2.3.2.3	. Matrix Spike and Matrix Spike Duplicate (MS/MSD)	30
	2.3.2.4	. Surrogates	30
3. A	ASSES	SMENT AND OVERSIGHT	30
4. I	DATA I	MANANGEMENT AND DOCUMENTATION	32
4.1.	W	P-QAPP	32
4.2.	Fir	nal Data Report	32
4.3.	La	boratory Documentation (Data Package Deliverables)	32
	4.3.1.	Data Package Deliverables	32
	4.3.2.	Electronic Data Reporting Formats	32
5. I	DATA :	REVIEW, VERIFICATION, AND VALIDATION	32
5.1.	Re	view of Data	33
5.2.	Da	ta Verification and Validation Stages	33
	5.2.1.	Stage 1	33
	5.2.2.	Stage 2A	34
	5.2.3.	Stage 2B	35
	5.2.4.	Stage 3	35
	5.2.5.	Stage 4	37
5.3.	Da	ta Verification and Validation Stages	37
5.4.	Us	ability Assessment	38
	5.4.1.	Precision	39
	5.4.2.	Accuracy	39
	5.4.3.	Representativeness	39
	5.4.4.	Completeness	39
	5.4.5.	Sensitivity	39
6. I	REFER	RENCES	39

LIST OF TABLES

Table 1. Project Organization and Distribution List	10
Table 2. Communication Pathways	11
Table 3. Analytical Laboratories, Contacts, and Analyses	13
Table 4. Project Quality Objectives	16
Table 5. Sample Locations, Media, Methods, Analytes of Interest, and Detection and Reporting Limits	18
Table 6. Sampling Summary (Number of Primary and Quality Control Samples) ¹	20
Table 7. Project Tasks	21
Table 8. Estimated Project Schedule	21
<u>LIST OF FIGURES</u>	
Figure 1. Project Organization Chart	9
Figure 2. Smallmouth bass	24
Figure 3. Bass sampling target collection areas, Site and Reference	25

APPENDICIES

Appendix A: Implementation Plan

Appendix B: Field Forms

Appendix C: Job Hazard Analysis (JHA)

Appendix D. PCB Congeners with Detection Limits and Reporting Limits

Appendix E. Dry ice sample packing and shipping

Appendix F. Laboratory Accreditation and Measurement Performance Criteria

Appendix G. Power Analysis

LIST OF ACRONYMS

μg/L microgram per liter
 ATL Acceptable Tissue Level
 CCB continuing calibration blank
 CCV continuing calibration verification

CoC chain of custody

CPR cardiopulmonary resuscitation

CTL Critical Tissue Level DL detection limit

DoD ELAP Department of Defense Environmental Laboratory Accreditation

DoD QSM Department of Defense Quality Systems Manual

DMC deuterated monitoring compounds

EDD electronic data deliverables

EPA United States Environmental Protection Agency
EMPC estimated maximum possible concentration
GC-MS gas chromatography mass spectroscopy

HAZWOPER Hazardous Waste Operations and Emergency Response

ICB initial calibration blank
ICV initial calibration verification

JHA Job Hazard Analysis
LCS laboratory control sample
mg/kg milligram per kilogram

MS matrix spike

MSD matrix spike duplicate

OU Operable Unit

PCB polychlorinated biphenyl PDT Project Delivery Team

POC point of contact PM Project Manager

PQO Project Quality Objectives

QC quality control

RI Remedial Investigation

RL reporting limit

SLV screening level value

SOP Standard Operating Procedure

SSHP Site Safety Health Plan
UCL upper confidence limit
UPL upper prediction limit

USACE United States Army Corps of Engineers

USGS United States Geological Survey

UFP-QAPP Uniform Federal Policy Quality Assurance Project Plan

WP-QAPP Work Plan with Quality Assurance Project Plan

1. PROJECT MANAGEMENT AND OBJECTIVES

1.1. Project Organization, Responsibilities and Authority

The Project Delivery Team (PDT) for this Work Plan with Quality Assurance Project Plan (WP-QAPP) includes members from the US Army Corps of Engineers (USACE) Portland and Seattle Districts as well as the US Geological Survey (USGS).

The project team provides the overall framework for the data collection approach by defining project objectives and data quality requirements, and ensuring that they are met during the execution of the project. USACE will obtain technical feedback from appropriate state and federal agencies and tribes and during ad hoc technical working group meeting(s), as needed. The roles of the project team members are described further in this section. Organization of the project is presented in Figure 1 and Table 1.

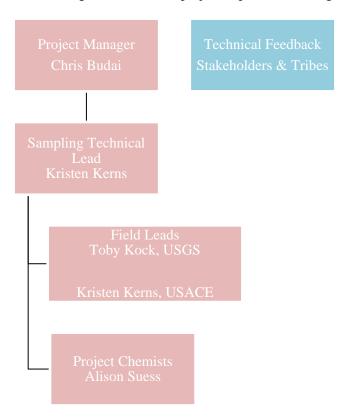


Figure 1. Project Organization Chart

Table 1. Project Organization and Distribution List

Personnel	Contact Information	Title
	USACE	
Chris Budai	333 SW 1st Ave Portland, OR 97204 Phone: 503-808-4725 Email: christine.m.budai@usace.army.mil	Project Manager
Bill Gardiner	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3322 William.W.Gardiner@usace.army.mil	Senior Technical Support
Alison M. Suess, Ph.D.	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3264 alison.m.suess@usace.army.mil	Project Chemist
Toby Kock	5501A Cook-Underwood Rd Cook, WA 98505 Phone: 509-538-2915 tkock@usgs.gov	Field Lead for USGS
Gabriel Hansen 5501A Cook-Underwood Rd Cook, WA 98505 Phone: 509-538-2915 ghansen@usgs.gov		Alternate Field Lead for USGS
Kristen Kerns	4735 E. Marginal Way S Seattle, WA 98134 phone: 206-764-3474 Kristen.kerns@usace.army.mil	Technical Lead/Field Lead for USACE

1.1.1. Communication Pathways

Communication is a key to the success of this project. Communication pathways describe the points of contact for resolving sampling and analysis problems, for distributing data to users, soliciting concurrence and obtaining approval between project personnel and contractors. Communication pathways are summarized in Table 2.

 Table 2. Communication Pathways

Communication Driver	Responsible Entity	Name Phone Number	Procedure (timing, pathway, etc.)
USACE management for this project Overall direction and Point of	Project Manager	Chris Budai	Assures that the overall direction of the project is consistent with USACE guidance
Contact for public		503-808-4725	Liaison with the Public
QAPP approval	Technical Lead	Kristen Kerns 206-764-3474	Coordinates with Project Manager, Project Lead, Chemist and Field Lead on project technical issues
Schedule, budget and technical issues			Reports to USACE PM regarding schedule, budget, and technical issues
Changes to schedule and budget			Notifies USACE PM of significant changes in execution or schedule
Oversight of final report Provides coordination among			Oversee USACE writing of final report and distribution to reviewers
team members			Provides input to QAPP and data
Ensures compliance with Site USGS Safety Plan and JHA (or another USACE representative)			reports Briefs field team on JHA and documents noncompliance
Delivery of samples to laboratory (or another USACE representative)			Coordinates with Project Chemist and laboratory for sample delivery
Writes QAPP with input from technical team members. Laboratory and data validation		Alison M. Suess, Ph.D.	Oversees writing of QAPP and Job Hazard Analysis (JHA) and ensures revision approval within agreed timeframe
	Project Chemists	206-764-3264	Oversees laboratory work
			Writes data validation report
			Provides laboratory and data validation components of QAPP
Provide direction to field teams on sample collections		Toby Kock / Gabe Hansen	Daily communication with team members during sampling events
Sampling activities summary	Field Lead	Kristen Kerns	Documents all field activities in Final Monitoring Report
			Coordinates with Project Chemist

1.1.2. USACE Personnel Responsibilities and Qualifications

USACE Project Manager

The project manager (PM), Chris Budai, is responsible for the execution of the scope, schedule, and budget for the Bradford Island project. She is the primary POC for communications with stakeholders. The USACE PM also has the authority to stop work of USACE staff. The USACE PM is the primary document controller for the WP.

USACE Technical Lead

The Technical Lead, Kristen Kerns, will oversee all activities of the USGS and USACE PDT, including quality assurance reviews, and maintain regular coordination to ensure adequate and timely flow of information for all work. The technical lead, or another USACE representative in the field, will serve as the site safety and health officer (SSHO) for this effort and coordinate daily field safety briefings.

USACE Project Chemists

The Project Chemist, Alison M. Suess, Ph.D. is directly responsible for laboratory coordination and matters related to chemistry. They are responsible for providing additional guidance to the Field Sampling Lead (Toby Kock / Gabe Hansen) in any matters relating to sampling, project chemistry and data quality.

Field Sampling Lead/Site Health and Safety Officer

Toby Kock/Gabe Hansen (USGS) and Kristen Kerns (USACE) are the designated field sampling leads. They are responsible for coordinating the sampling with relevant Bonneville Project staff and execution of sampling. They may communicate directly with the PM, Technical Lead, and Project Chemists as needed during the field sampling event.

Special Training Requirements and Certifications

Project staff shall be qualified to perform their assigned jobs. Field sampling personnel conducting or monitoring sampling activities are to be trained by the field sampling lead in accordance with established USACE protocols.

Field Staff

All project staff participating in on-site field activities shall have current HAZWOPER training in accordance with 29 Code of Federal Regulations (CFR) Part 1910.120, or be directly supervised by personnel with current HAZWOPER training. The technical lead and/or field sampling lead has HAZWOPER training in accordance with the same standard as well as a current certification in first aid and CPR. All field personnel responsible for packing and shipping samples using dry ice also have training and certification in accordance with 49 CFR 172.704 and the IATA Dangerous Goods regulation.

Laboratory Contact

The analytical laboratories and applicable information that will be used for this project are listed below. In Table 3.

Table 3. Analytical Laboratories, Contacts, and Analyses

Lab Name and Sample Type	Lab Address	POC	Contact Info	Role
Eurofins Lancaster Laboratories Environmental Testing LCC Bass and Bait Samples	2425 New Holland Pike Lancaster, PA 17601	Tim Witrzek	847-324-3320	Federal Program Manager, Prime Contractor, EMT

1.1.3. External Technical Review Personnel Responsibilities and Qualifications

State and federal agencies and tribes represent their respective agencies and provide technical review of the QAPP.

1.2. Project Planning

1.2.1. Project Planning (Scoping)

Several planning meetings were held within USACE and with external technical reviewers during development of the initial sampling effort for bass in fall 2020 as well as this subsequent sampling effort. Topics discussed in those meetings include:

- Schedule
- Sampling Design and Data Collection
- Analytes

The outcomes of the meetings are documented by incorporation into the initial WP-QAPP supporting the fall 2020 sampling effort and again in this WP-QAPP.

1.2.2. Problem Definition, Site History, and Background

USACE conducted a Remedial Investigation and draft Feasibility Study for the in water portion of Bradford Island, known as the River Operable Unit (OU), in accordance with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) of 1980 Executive Order 12580. As part of the Feasibility Study process, USACE conducted a baseline risk assessment, which found unacceptable risk to human health and the environment from exposure to PCB contaminated sediment in the River OU.

Field efforts performed between 2006 and 2011 in support of the Remedial Investigation sampled smallmouth bass and found elevated levels of PCBs in some of these fish. PCBs in crayfish tissues from

the Site were also elevated, relative to the reference area. During the feasibility study, USACE conducted supplemental passive porewater sampling and sediment trap deployment in 2017 and 2018. This sampling effort included underwater video survey, with underwater images of the river bottom along the northern shoreline of Bradford Island showing minimal sediment and large cobbles and boulders. This lack of sediment raised concern regarding the continued presence of contaminated sediment and the validity of the CSM developed in support of the FS. Subsequently, USACE began collecting data to update the CSM for the River OU. The intent of this data is to help inform the current site conditions for the River OU to aid in development of remedial action alternatives in the feasibility study

In 2020, USACE conducted additional in-situ porewater sampling to better understand the location of potential primary source contamination along the northern shoreline of Bradford Island. As part of the reevaluation and update to the CSM, tissues of fish and invertebrates are being sampled for chemical analysis. Bass represent an important resident prey species for human health via the fish ingestion pathway.

This QAPP provides the approach and methods for sampling and analysis of bass. Smallmouth bass tissue and tracking efforts were completed in fall of 2020. This field effort for spring of 2022 is intended to provide information on potential seasonal variability in tissue chemical concentrations and movement of smallmouth bass. Seasonal variability for several parameters, including overall body mass, relative liver mass, lipid content, and the reproductive life cycle is a documented occurrence for smallmouth bass in the Columbia River (Rose, et.al., 2013). Smallmouth bass are semi-dormant during the winter when water temperatures are low. As temperatures increase during the spring, feeding and migration increase. These factors could reasonably be anticipated to influence chemical body burden in smallmouth bass near Bonneville Dam. The intent of this sampling effort is to mirror the previous methods and analyses performed in the fall of 2020 and associated QAPP (USACE, 2020) to allow for this seasonal comparison.

USACE has contracted with the USGS to collect smallmouth bass samples for tissue analysis and tagging and to evaluate the movements of smallmouth bass near Bonneville Dam using acoustic telemetry.

1.3. Project Quality Objectives and Measurement Performance Criteria

1.3.1. Development of Project Quality Objectives Using the Systematic Planning Process

Project Quality Objectives (PQOs) are developed through the systematic planning process as described in the UFP-QAPP Guidance. PQOs specify the type, quantity, and quality of data needed to ensure that project data can be used for the intended purpose to answer specific environmental questions, support environmental decisions, and determine technical activities that will be conducted. The PQOs developed for this project are described in Table 4.

The overall goal of this tissue collection effort and telemetry is to update and confirm the conceptual site model presented in the Remedial Investigation. Smallmouth bass tissue and tracking efforts were completed in fall 2020. This field effort for spring of 2022 is intended to provide information on potential seasonal variability in tissue chemical concentrations and movement of smallmouth bass. The intent of this sampling effort is to mirror the previous methods and analyses performed in the fall of 2020 and associated QAPP (USACE, 2020) to allow for this seasonal comparison. The results of this data will be

looked at comprehensively with other data associated with clam tissue, passive sampling, and future sediment sampling. This data may be used to update the risk assessment and provide current risk communications to tribal and recreational fishers in the area. The intent is not to redo the baseline risk assessments but supplement the dataset to reflect current conditions.

The analytes for tissues were selected based on their high contribution to Site risks. PCBs provide a direct indication of historical contamination at Bradford Island from the disposal of PCB containing transformers. PCB contamination has historically been identified in every sampled media at the site and also contributes a majority of risk to both ecological and human health receptors. Organochlorine pesticides were identified for analysis in tissue based on concentrations in bass tissue that contributed a notable fraction to overall risk. However, there is uncertainty if the elevated concentrations are attributable to site exposures or the result of matrix interferences during analysis. As such, analysis for organochlorine pesticides for this field effort will help to confirm its role in risk. Lastly, mercury is ubiquitous at elevated concentrations throughout this portion of the Columbia River. However, given previous industrial activities as the site and associated risk, current mercury concentrations will be evaluated as part of this effort. The goal of this study is not to decern which factors influence contaminant body burden, but rather to determine if spring conditions influence body burden of the spring subpopulation. Should the spring dataset prove to be statistically similar to the fall dataset, these two sampling efforts will collectively contribute to a larger dataset that can further assist in site characterization and risk communication.

PQOs one through three are identical to the PQOs in the QAPP for the smallmouth bass sampling effort that took place in the fall of 2020 (USACE, 2020). PQOs four and five unique to this QAPP, aimed at understanding seasonal differences between the spring and the fall between tissue concentrations (PQO-4) and movement patterns (PQO-5) for smallmouth bass. All statistical analysis will rely on use of ProUCL statistical software, Version 5.1.

 Table 4. Project Quality Objectives

Step 1:	Step 2:	Step 3:	Step 4:	Step 5:	Step 6:	Step 7:
State the Problem	Identify the Goals of	Identify Information Inputs	Define the Boundaries of	Develop the	Specify Performance or	Develop the Detailed Plan
	the Study		the Study	Analytic Approach	Acceptance Criteria	for Obtaining Data
1) Are there any significant differences in River OU (Site) bass concentrations relative to reference concentrations?	Evaluate differences between tissue concentrations at the Site versus reference area. Understand site concentrations and magnitude of impacts from the site relative to concentrations representative of unimpacted receptors. Update and reconfirm conceptual site model.	The evaluation will use results from the analysis of samples collected in the Site and analysis of samples representative of reference concentrations. Reference concentrations for bass will be determined by fish collected near Bonneville Dam that are from a separate population than those bass impacted by contamination from Bradford Island. Bass collected from previous sampling efforts (2011 and earlier) that represent reference population concentrations will also be compared. Information from other sampling efforts for bass in the Columbia River may also be considered.	Tissue samples will be analyzed for the analytes of interest. For bass, sample locations will focus on the northern shoreline of Bradford Island, Goose Island, and the Forebay up to RM 147.	Statistical comparison between Site versus reference value(s) to determine significant differences. Visual evaluation of data and statistical outlier test.	See Data Usability Assessment (Section 5.1).	See Sampling Design, Location, and Methods (Sections 2.1).
2) Are there any changes in tissue concentrations for bass collected from the Site over time?	Evaluate changes in tissue concentrations of target analytes at the Site for bass and crayfish collected during 2006 (Site), 2007/2008 (reference), and 2011 and tissue collected in 2020. Confirm current conditions relative to previous information in order to update the conceptual site model.	The evaluation will use results from the analysis of samples collected in the Site in 2020 relative to samples collected between 2006 and 2011. Potential temporal changes for the reference concentrations/area will also be assessed.	Tissue samples will be analyzed for analytes of interest. Sample locations will focus on the Site and reference concentrations/area. Historic data includes collection efforts in 2006 (Site), 2007/2008 (reference), and 2011 relative to the 2020 sampling effort.	Statistical comparison for data collected over time, both RI and post RI data.	See Data Usability Assessment (Section 5.1).	See Sampling Design, Location, and Methods (Sections 2.1).
3) Where are bass potentially exposed to PCB contaminated sediment in the Site? How do bass move through different parts of the Site? How do bass move between different areas of the site, including the north shore of Bradford Island and Goose Island?	Evaluate movement of bass as an indicator of where PCB exposure may occur.	The evaluation will use results from acoustic telemetry of approximately 40 smallmouth bass tracked in the Site.	Bass within the Site will be tracked with acoustic telemetry. Initial capture locations for tagging will be focused in the Site.	Telemetry data analyzed using SAS Statistical Software.	See Data Usability Assessment (Section 5.1).	See Sampling Design, Location, and Methods (Sections 2.1).

Step 1: State the Problem	Step 2: Identify the Goals of the Study	Step 3: Identify Information Inputs	Step 4: Define the Boundaries of the Study	Step 5: Develop the Analytic Approach	Step 6: Specify Performance or Acceptance Criteria	Step 7: Develop the Detailed Plan for Obtaining Data
	T		T	T -: -	T	T
4) Are there seasonal differences in bass tissue concentrations at the Site and Reference Area?	Evaluate potential seasonal differences between tissue concentrations at the Site and reference area during the spring relative to concentrations from the previous sampling effort in August and September 2020.	The evaluation will use results from the analysis of samples collected in the Site and analysis of samples representative of reference concentrations. Additional variables including length, weight, and lipid content will be accounted for. Reference concentrations for bass will be determined by fish collected near Bonneville Dam that are from a separate population than those bass impacted by contamination from Bradford Island. Bass collected from previous sampling efforts (2011 and earlier) that represent reference population concentrations will also be compared. Information from other sampling efforts for bass in the Columbia River may also be considered.	Tissue samples will be analyzed for the analytes of interest. For bass, sample locations will focus on the northern shoreline of Bradford Island, Goose Island, and the Forebay up to RM 147.	Statistical comparison between fall versus spring value(s) to determine significant differences. Visual evaluation of data and statistical outlier test.	See Data Usability Assessment (Section 5.1).	See Sampling Design, Location, and Methods (Sections 2.1).
5) Are there seasonal differences in movement of bass through different parts of the site? Does seasonal variability impact how bass move between different areas of the site, including the north shore of Bradford Island and Goose Island?	Evaluate movement of bass as an indicator of where PCB exposure may occur during the spring relative to previous sampling conducted in August and September 2020.	The evaluation will use results from acoustic telemetry of approximately 40 smallmouth bass tracked in the Site.	Bass within the Site will be tracked with acoustic telemetry. Initial capture locations for tagging will be focused in the Site.	Telemetry data analyzed using SAS Statistical Software.	See Data Usability Assessment (Section 5.1).	See Sampling Design, Location, and Methods (Sections 2.1).

Table 5. Sample Locations, Media, Methods, Analytes of Interest, and Detection and Reporting Limits

Sample Locations and Media Site and Reference Area Bass Tissue; Bait	Method PCB Congeners, EPA 1668C	Analytes 209 PCB congeners (µg/kg)	7 Tissue DL 0.001-0.00907	0.001- 0.096	SLVs for Subsistence Fishers ¹	CTLs for Fish & Shellfish Exposed to Bass Tissue ¹	ATLs for Individual Birds Exposed to Bass Tissue ¹	ATLs for Individual Mammals Exposed to Bass Tissue ¹
		2,4'-DDD (μg/kg)	0.660	3.40	3.4	54	13	580
		2,4'-DDE (μg/kg)	0.660	3.40	3.4	54	13	580
		2,4'-DDT (μg/kg)	0.660	3.40	3.4	54	13	580
		4,4'-DDD (μg/kg)	0.660	3.40	3.4	54	13	580
		4,4'-DDE (μg/kg)	0.660 1.58	3.40	3.4	54 54	13 13	580 580
		4,4'-DDT (µg/kg)		3.40	0.72			
		alpha-BHC(µg/kg)	0.340	1.70		NA	NA NA	NA
		beta-BHC (μg/kg)	0.880	2.00	0.72	NA	NA	NA
		delta-BHC (μg/kg)	0.900	2.00	0.72	NA	NA	NA
Site and	Organochlorine	gamma-BHC (µg/kg)	0.420	1.66	0.72	NA	NA	NA
Reference Area Bass	Pesticides, EPA 8081	alpha-Chlordane (cis) (µg/kg)	0.340	1.70	3.3	60	1,200	3,300
Tissue; Bait		gamma-Chlordane (trans) (µg/kg)	0.500	1.66	3.3	60	1,200	3,300
		Dieldrin (µg/kg)	0.660	3.40	0.072	260	44	150
		Endosulfan I (µg/kg)	0.440	1.66	NA	NA	NA	NA
		Endosulfan II (µg/kg)	2.20	4.60	NA	NA	NA	NA
		Endrin (µg/kg)	1.36	3.40	NA	NA	NA	NA
		Endrin Aldehyde (µg/kg)	0.660	3.40	NA	NA	NA	NA
		Methoxychlor (μg/kg)	3.60	13.4	NA	NA	NA	NA
Site and Reference Area Bass Tissue; Bait	Mercury, EPA 7471	Mercury (mg/kg)	0.025	0.06	0.049	0.088	0.074	0.12
Site and Reference Area Bass Tissue; Bait	Total Lipids, Sulfo- Phospho- Vanillin Colorimetric Method (Van Handel 1985)	Total Lipids	0.05%	0.2%	NA	NA	NA	NA

1. DEQ 2007. Guidance for Evaluation of Bioaccumulative Chemicals of Concern in Sediment. Final. January 31 (see Appendix J in the RI [URS. 2012. Upland and River Operable Units Remedial Investigation Report. Bradford Island, Bonneville Dam Forebay, Cascade Locks, Oregon. June]).

ATL=Acceptable Tissue Level

CTL=Critical Tissue Level

SLV=Screening Level

Matrix	Location	Target Num. Per Location	Analyses	Primary Samples	Field Duplicate Samples ²	MS/MSD ³	Total Number of Field Samples
	Bradford Is.	40 (20 min)	PCB Congeners	80	8	4/4	96
Site and Reference Bass Tissue	Goose Is.	20	Organochlorine Pesticides	80	8	4/4	96
Tissue	Reference	20	Mercury	80	8	4/4	96
	Reference	20	Total Lipids	80	8	0	88
			PCB Congener	2	1	1/1	5
Bait (for bass)	NA	NA	Organochlorine Pesticides	2	1	1/1	5

Table 6. Sampling Summary (Number of Primary and Quality Control Samples)¹

Mercury

1.3.2. Measurement Performance Criteria

Performance criteria specify the acceptable levels of uncertainty in measured data that can be used to support project decisions and achieve PQOs. Performance criteria for the analytical methods are specified in the laboratory procedures and are compliant with current DoD QSM unless otherwise noted. Any data which fall outside of these criteria must be justified, and the effects on decisions must be assessed.

1.4. Secondary Data Evaluation

Daily water temperature from the Bonneville Dam Forebay will be obtained from the Columbia Basin Research DART River Environment Daily Data (http://www.cbr.washington.edu/dart/query/river_daily) for the time period corresponding to the field effort.

1.5. Project Overview and Schedule

Through project planning, the project team has agreed on the purpose of the project, the environmental questions that are being asked, and the environmental decisions that must be made. Table 7 provides a summary of the project tasks to be completed and Table 8 describes the project schedule. The field schedule is partially dictated by spill operations at Bonneville Dam. The northern shoreline of Bradford Island is within the portion of the forebay designated as a Boating Restriction Zone (BRZ). During spill operations, no boat traffic is permitted within this portion of the site. Thus, sample collection in the BRZ is limited to the months of September to 10 April.

^{1.} Does not include laboratory quality control samples such as laboratory duplicates and control spikes. The mass required provided by the laboratory and listed in Table 10 includes sufficient mass for all field and laboratory quality control samples.

^{2.} Field duplicate samples will be collected at a rate of 1 per 10 primary samples.

^{3.} MS/MSD samples will be collected at a rate of 1 pair per 20 primary samples.

Table 7. Project Tasks

Plan, Prepare WP-QAPP & Obtain Laboratory Quote

Prepare and finalize WP-QAPP; obtain laboratory quotes.

Sampling Tasks

- Collect site and reference area bass
- Tag bass for acoustic telemetry

Analytical Tasks

- Chemical analysis of bass tissue
- Data collection and analysis of acoustic telemetry

Quality Control Tasks

Chemical analytical methods QC will comply with DoD QSM or laboratory SOPs as applicable.

Secondary Data

No secondary data will be collected.

Data Management Tasks

- Project Chemists will review and store analytical chemistry data.
- USGS will review and store acoustic telemetry data.

Documentation and Records

- Field notes will be recorded in a field notebook or on field log sampling sheets, then scanned and electronically stored.
- Field notes will contain the following: date and time of sample collection, weather conditions, sample identification number, type of sample, lure/bait, length, mass, any procedural steps taken that deviate from those outlined in this WP-QAPP.
- Laboratory analytical results will be stored.

Data Validation and Data Packages

• 100% of chemistry data packages will be validated through Stage 2A by the Project Chemists. A subset of data (10%) will undergo Stage 4 data validation All data packages will be delivered in sufficient detail to support a Stage 4data validation.

Data Review Tasks

- The laboratory performing chemical analyses of samples will verify that all data are complete for samples received.
- Chemical data will be validated.
- Validated data will be reviewed.
- Data usability will be assessed.
- Measurement performance criteria set in WP-QAPP will be checked.
- Data limitations will be determined. Data will be compared to PQOs.

Table 8. Estimated Project Schedule

Task #: Description	Start	Finish					
Task #1: Plan, Prepare WP-QAPP and Obtain Laboratory Quotes							
Prepare Draft WP-QAPP	3 January 2022	28 January 2022					
External Review	31 January 2022	18 February 2022					
Finalize WP-QAPP	21 February 2022	4 March 2022					
Obtain laboratory quote, finalize, and receive sample containers	31 January 2022	1 March 2022					
Purchase Field Equipment	31 January 2022	1 March 2022					
Task #2: Field Work							
Area within BRZ (BRZ permit required; spillway opens 10 April)	7 March 2022	10 April 2022					
Area outside BRZ	7 March 2022	30 April 2022					
Task #3: Review Data and Prepare Report							

Task #: Description	Start	Finish	
Receive Data Deliverable from Lab	1 May 2022	30 June 2022	
Data Validation	1 July 2022	1 August 2022	
Receive Data from USGS for Acoustic Telemetry		30 August 2022	
Draft and Final Data Reports	1 August 2022	31 October 2023	

2. DATA GENERATION AND ACQUISITION

2.1. Sampling Tasks

Sample identification and field sampling will be performed following the protocols described in this section. Contingencies may arise during activities that will require modification of the general procedures outlined herein. Such modifications will be at the discretion of the field lead after consultation with the study technical lead and PM, the boat captain, and sampling team in the field. All modifications will be recorded and document in the field or data report, as appropriate.

2.1.1. Sampling Process Design and Rationale

The USGS will be leading the sample collection effort for both tissue collection for chemical analysis as well as capture and tagging of smallmouth bass. Appendix A provides the implementation plan for those field sampling efforts. USACE staff will be on site to support the USGS, particularly for processing of tissue for shipment to the laboratory for chemical analysis.

Reference Tissue

For smallmouth bass, both Site and reference bass will be collected in the immediate area of Bonneville Dam. The intent is to increase potential catch numbers in the area closest to Bradford Island. Based on previous sampling conducted in 2011 and earlier, it is possible that two distinct populations of bass are present in the Bonneville dam area; those exhibiting contamination likely obtained from Bradford Island and those not/less impacted by contamination at Bradford Island. See section 2.1.2 for additional information.

Given the approach to collect reference tissue for bass in the same general vicinity as Site fish impacted by Bradford Island, the results will need to be evaluated both statistically, visually, and against existing datasets representative of reference or background concentrations. ProUCL will be used to visually represent the data and statistically evaluate the dataset for outliers. Any outliers are assumed to be representative of impacts from Bradford Island contamination. Based on previous collection efforts, it is possible that bass of elevated concentration will be captured near Goose Island. While areas of collection are not necessarily indicative of the source of contamination for bass, fish captured from Goose Island will initially be evaluated separately from the Bradford Island bass. If telemetry data indicate frequent movements from Bradford Island to the Goose Island area or if other media indicate there are no contamination sources from the Goose Island area – the interactions between the two areas will be evaluated. Previous datasets associated with Bradford Island fish collection and other nearby fish collection studies in the Columbia River will also be referenced to identify concentrations that

appropriately represent a reference concentration. Bass collected as part of this field effort will be statistically compared to those reference concentrations.

2.1.2. Sample Collection Procedures

Sample collection will be led by the USGS. An Implementation Plan describing collection procedures for smallmouth bass is included in Appendix A. Collection procedures are identical to those previously used.

The intent is to prioritize bass collection for chemical analysis of tissue first, then collect bass for acoustic tagging once all fish are captured for tissue analysis. However, given the potential for lower catch rates during the spring relative to previous fall catch rates, there may be a need to alternate between collection of fish for tissue analysis and acoustic tagging to ensure both objectives are met. The goal will be to collect 80 fish for tissue analysis and 40 fish for acoustic telemetry. However, if catch rates are diminished, a total of 60 fish for tissue analysis is considered acceptable (20 from Bradford, 20 from Goose Island, 20 from Reference). The most likely instance to encounter reduced catch rates is prior to the start of spill on April 10th. This would most likely impact the catch rates within the targeted Bradford Island catch area of interest. A total of 40 fish are required for acoustic telemetry tagging.

Target species for capture is the smallmouth bass. Sexually mature bass are typically represented by a total length greater than 150mm. Bass of this size will be targeted for chemical sampling and telemetry. However, bass out of this range may also be retained, especially if abundance is low. An effort will be made to tag bass proportionally throughout the size range. Total mass of each individual fish collected for chemical analysis will also be recorded.

Gastric lavage will be performed on all bass captured subject to chemical analysis to eliminate potential influence of stomach content to analytical results. Stomach content will be captured in a sieve and retained from individual fish if sufficient mass is collected (minimum 40g). Stomach content will be archived for 1 year at 4°C for potential future chemical analysis. The remaining whole body of each fish (excluding stomach contents) will be wrapped in aluminum foil, double bagged, and shipped to the laboratory, where it will be homogenized prior to analysis. This is the same processing method that was performed on the fish from the fall 2020 sampling effort. For field duplicates, the laboratory will homogenize the sample and then split the homogenate into a primary sample and a field duplicate sample prior to analysis.

Non-target species captured via angling will be document, identified as juvenile or adult, then released with minimal handling. If sculpin are incidentally captured, they will be retained and archived for potential future chemical analysis.

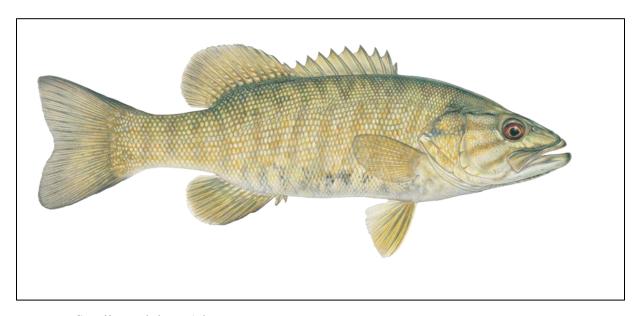


Figure 2. Smallmouth bass (photo source: https://www.dfw.state.or.us/)

Bradford Island and Reference Sampling Locations

Target collection locations for angling are along the northern shoreline of Bradford Island, in the vicinity of Goose Island, and in the forebay immediately adjacent and upstream of those areas. The map (Figure 3) indicates the areas of focus for angling efforts and the targeted number of smallmouth bass in each of those areas. However, fishing effort may be adjusted based on the locations of fish and catch success in the event that targeted numbers cannot be achieved. Information from historic collection efforts will be used to help guide staff to where successful collection previously occurred.

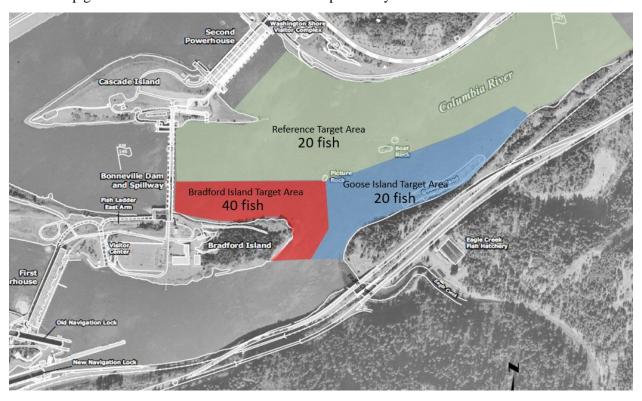


Figure 3. Bass sampling target collection areas, Site and Reference

Bait

The intent is to use lures as the primary means of catch for bass. However, bait will be available as a backup. Before use, a representative samples of bait (worms) for bass collection will be analyzed for PCB Congeners, organochlorine pesticides, and total mercury. Because methods with low-level detection limits are proposed, it is possible that detections of analytes will be reported. Bait will not be omitted if detections are reported, and analytical results will be provided for informational purposes only. Application of the analytical results for bait will be most relevant if retained stomach content is analyzed at a later date.

Chemical Analysis

For chemical analysis, smallmouth bass will be analyzed as individual samples, and no compositing is anticipated. All specimen will be wrapped in aluminum foil, double bagged, labeled, and placed on dry ice for shipment to the laboratory. The goal is to collect 80 smallmouth bass for chemical analysis. Statistical analysis to support the target collection numbers is based on the previous QAPP (see Appendix D of previous QAPP, USACE, 2020).

2.1.3. Sample Naming Convention

Bass will be given an identification for each sample. The naming convention will include initials for the specimen type (SB=smallmouth bass, a number indicating the boat crew (4, 5, 6 etc.), and a 3-digit sample number (001, 002, 003, etc.). The number indicating the boat crew will follow sequentially with the previous sampling effort, thus boat crews for this round of sampling will start with "4". Field duplicate samples will end in "FD", and matrix spike and matrix spike duplicate samples will end in "MS" and "MSD", respectively.

Examples:

SB4001 (primary sample)

SB4001FD (field duplicate associated with primary sample #1)

SB4001MS (matrix spike associated with primary sample #1)

SB4001MSD (matrix spike duplicate associated with primary sample #1)

Table 10. Methods, Sample Containers, Volumes, Preservation, and Holding Times for Smallmouth Bass Tissue and Bait Samples

Analytes	Analytical Method	Container Type/Quantity	Preservation	Minimum Mass per Sample ¹ (g)	Holding Time
PCB congeners	EPA 1668C	Aluminum foil inside Ziploc bag	Thawed: 4 °C ± 2 °C Frozen: -20 °C	40	Thawed: 14 days Frozen: 1 year
Organochlorine Pesticides	EPA 8081	Aluminum foil inside Ziploc bag	Thawed: 4 °C ± 2 °C Frozen: -20 °C	30	Thawed: 14 days Frozen: 1 year
Mercury	EPA 7474	Aluminum foil inside Ziploc bag	Thawed: 4 °C ± 2 °C Frozen: -20 °C	6	Thawed: 14 days Frozen: 1 year
Total Lipids	Sulfo-Phospho- Vanillin Colorimetric Method (Van Handel 1985)	Aluminum foil inside Ziploc bag	Thawed: 4 °C ± 2 °C Frozen: -20 °C	3	Thawed: 14 days Frozen: 1 year

^{1.} Tissue mass listed includes all laboratory and field quality control samples, such as blank, duplicate, LCS/LCSD, MS/MSD, and potential re-extraction.

2.1.4. Decontamination Procedures

All potential sources of contamination in the field will be identified by the field lead, and appropriate steps will be taken to minimize or eliminate contamination. Ice chests will be scrubbed clean with Alconox® or Liquinox® detergent and rinsed with distilled water after use to prevent potential cross contamination. To avoid contamination from melting ice, the dry ice will be separated from samples by placing all samples in large plastic bags. Prior to each use, sampling equipment will be cleaned with Alconox® or Liquinox® phosphate-free detergent and rinsed with deionized water.

2.1.5. Field Equipment Calibration, Maintenance, Testing and Inspection Procedures

No field equipment requires calibration, maintenance, testing and inspection. If any sampling procedures are changed to include use of field equipment, that information will be included in the field notes.

2.1.6. Supply Inspection and Acceptance Procedures

Inspection and acceptance of supplies and consumables will be conducted prior to field work in order to ensure that the appropriate type and quantity of supplies are brought to the field. Any supplies and consumables used in the sample collection process or instrument calibration will be inspected.

2.1.7. Field Documentation Procedures

Field documentation provides a permanent record of field activities and can be used, if necessary, to trace possible introduction of field sampling error.

Field notes will be maintained either in a bound logbook, or on field sampling log sheets. After fieldwork is complete, electronic copies will be made of the field notes and the electronic copies will be stored in the project files. All information pertinent to the sampling effort will be recorded in the field notes.

Documentation in the field notes will be at a level of detail sufficient to explain and reconstruct field activities without relying on recollection by the field team members. The Field Sampling Lead has overall responsibility for accuracy and completeness of field notes. Each page/form will be consecutively numbered. All entries will be made in indelible ink and corrections will consist of lined-out deletions. As a minimum, the applicable items for the entry into the field notes are listed below.

General Information

- Date
- Time
- Weather conditions
- Names of personnel present

Sampling Information

- Location of sample
- Type of sample
- Sample identification number
- Associated QC samples
- Any unusual observations

2.1.8. Sample Delivery

Sample delivery procedures include packaging, labeling, and shipment to the laboratory. These procedures are designed (1) to preserve sample quality so that analyses will yield results representative of site conditions, (2) to protect and inform sample handlers, including shippers and laboratory personnel, and (3) to provide a paper trail to allow cross referencing of sample collection locations with analytical results. See Appendix E for dry ice sampling packing and shipping methods.

All samples will be shipped on dry ice. Dry ice will be supplied by the following vendor:

OXARC® Inc. 19310 NE San Rafael St, Portland, OR 97230 (503) 618-1625

Samples will be shipped from the nearest FedEx facility that accepts packages containing dry ice:

FedEx Ship Center 5159 NE Cornfoot Rd Portland, OR 97218

All samples will be labeled with its own sample identification number and all other applicable information. Samples will be shipped with dry ice overnight via FedEx to the laboratory. To avoid potential shipping delays, shipments for Thursday and Friday will be avoided and held in a freezer or on dry ice till the following Monday for shipment. The shipping address for the laboratory is:

Laboratory - TBD Street address City, state, zip code

2.1.9. Sample Custody

A sample is in "custody" if it is in the actual physical possession of authorized personnel or in a secure area that is restricted to authorized personnel. Custody procedures ensure data authenticity and defensibility. Chain of custody (CoC) forms will accompany sample containers during transit to the laboratory and be checked by the laboratory upon receipt.

2.2. Analytical Tasks

Once samples have been collected, they will be analyzed by the laboratories. The Project Chemists will validate the analytical data.

The following sections address all components of project-specific analytical measurements; method and laboratory-specific QC measurements; acceptance criteria; corrective actions; calibration procedures; equipment and supply maintenance; testing; and inspection requirements. Modifications to approved procedures, alternate procedures, or additional procedures are to be pre-approved in writing by the Project Chemist.

2.2.1. Analytical Methods

See Table 5 for analytical methods that will be used for analysis of tissue samples.

2.2.2. Analytical Instrument Calibration Procedures

Calibration procedures and instrumentation shall be consistent with the requirements of the methods.

2.2.3. Analytical Instrument and Equipment Maintenance, Testing, and Inspection Procedures

Maintenance, testing, and inspection procedures shall be consistent with the requirements of the methods.

2.3. Quality Control Samples

Quality control (QC) samples are collected and analyzed for the purpose of assessing the quality of the sampling and analysis performed by the field personnel and the primary laboratory. The Project Chemist will coordinate selection of QC samples prior to each sampling event.

2.3.1. Field Quality Control Samples

2.3.1.1. Field Duplicates

Field duplicate samples will be collected at a rate of 1 per 10 primary samples. Field duplicate samples for tissue will be evaluated at 50% relative percent difference.

2.3.1.2. Trip Blanks

No trip blanks will be collected for this sampling event as they are not necessary for the selected methods.

2.3.1.3. Equipment Rinse Blanks

No equipment rinse blanks will be collected since there is no reusable sampling equipment such as scoops or containers utilized in bass collection.

2.3.2. Analytical Method Quality Control Samples

Method QC includes the analyses and activities required to ensure that the analytical system is in control prior to and during an analytical run. Method QC requirements for this project include the following: method blanks, surrogate spikes, matrix spikes/matrix spike duplicate pairs, and laboratory control samples.

2.3.2.1. Method Blanks

Method blanks are composed of organic/analyte-free water processed simultaneously with and under the same conditions as samples through all steps of the analytical procedure. Method blanks verify that the measurement system is free of contamination.

2.3.2.2. Laboratory Control Samples (LCS)

Laboratory control sample (LCSs) are composed of organic/analyte-free water spiked with verified amounts of analytes. They are used to evaluate accuracy and precision, including to establish intralaboratory or analyst-specific precision or to assess the performance of all or a portion of the measurement system. The LCS is analyzed in the same manner as a sample, including preservation. Laboratory acceptance criteria will be used for evaluation of the results.

2.3.2.3. Matrix Spike and Matrix Spike Duplicate (MS/MSD)

MS/MSD samples are used to evaluate matrix interference and to determine laboratory accuracy and precision. For methods that require MS/MSDs, MS/MSD samples will be collected at a rate of 1 pair per 20 primary samples. Laboratory acceptance criteria will be used for evaluation of the results.

2.3.2.4. Surrogates

Surrogates are substances with properties that mimic the analyte of interest. A surrogate is unlikely to be found in environment samples, and is therefore added to assess accuracy of the results. Laboratory acceptance criteria will be used for evaluation of the results.

3. ASSESSMENT AND OVERSIGHT

Laboratory and field operations have established policies and procedures, and they designate authorities for implementing corrective action when nonconforming work or departures from the policies and

procedures in the quality system or technical operations have been identified. Both field and laboratory operations shall follow all corrective action requirements in methods and SOPs.

The following laboratory documentation is to be made accessible to the USACE Project Chemist. Corrective actions may be required, at the request of USACE, for the following conditions:

- Laboratory Procedures
- QC data outside the defined acceptance windows for precision or accuracy
- Blanks or LCS's that contain contaminants above acceptable levels stated in the Project Quality Objectives
- Undesirable trends in spike or surrogate recoveries or RPD between spiked duplicates
- Unusual changes in method detection limits
- Deficiencies identified during internal or external audits or from the results of performance

The following corrective actions should be taken for common problems:

Incoming Samples - Problems noted during sample receipt are to be documented. The USACE Project Chemist is to be notified for problem resolution.

Sample Holding Times - If a maximum holding time is or may be exceeded by the laboratory, the USACE Project Chemist must be notified for problem resolution. The USACE Project Chemists may require re-sampling for the requested parameters.

Instrument Calibration - Sample analysis may not proceed until initial calibrations meet method criteria. Calibrations must meet method time requirements or recalibration must be performed. Continuing calibrations that do not meet accuracy criteria should result in a review of the calibration, rerun of the appropriate calibration standards, and reanalysis of samples affected back to the previous acceptable calibration check.

Limit of Quantitation (LOQ) - Appropriate sample clean-up procedures must be employed to attempt to achieve the practical quantitation limits as stated in the method. If difficulties arise in achieving these limits due to a particular sample matrix, the laboratory should notify the USACE Project Chemists of the problem for resolution. Dilutions are to be documented in the case narrative along with the revised practical quantitation limits for those analytes directly affected. Analytes detected above the method detection limits (MDLs) but below the practical limit(s) of quantitation are to be reported as estimated values and qualified "J".

Estimated maximum possible concentration (EMPC-qualified) values will be treated as detected concentrations. EMPC-qualification is used when mass spectrometry results meet all of the identification criteria in the method except the ion abundance ratio criteria.

Method Quality Control - Results related to method QC, including blank contamination, duplicate measurement reproducibility, MS/MSD recoveries, surrogate recoveries, LCS recoveries, and other method-specified QC measures are to meet the laboratory's SOPs and PQOs specified in this plan. Otherwise, the affected samples may be reanalyzed and/or re-extracted and reanalyzed within method-required holding times to verify the presence or absence of matrix effects. In order to confirm matrix

effects, QC results must observe the same direction and magnitude (ten times) bias. The USACE Project Chemist should be notified as soon as possible to discuss appropriate corrective action.

Calculation Errors - Reports must be reissued if calculation and/or reporting errors are noted with any given data package. The case narrative is to state the reason(s) for re-issuance of a report.

4. DATA MANANGEMENT AND DOCUMENTATION

4.1. WP-QAPP

An electronic copy of the WP-QAPP (including appendices) will be stored in USACE project files and provided to the Technical Advisory Group.

4.2. Final Data Report

Upon completion of the sampling event and receipt/review of the validated data, USACE will prepare a final data report. The report will include the following:

- Narrative and timeline of project activities
- Summary of sampling, chemical testing, and any deviations from the QAPP
- Analytical data summary and discussion
- Figures, tables, and appendices

The appendices will include field logs, laboratory analytical reports, data validation reports, and data summary tables with associated validation flags.

4.3. Laboratory Documentation (Data Package Deliverables)

4.3.1. Data Package Deliverables

The analytical data packages from the laboratories will be provided to the Project Chemist in sufficient detail for the required level of data validation. The analytical data packages will be validated to Stage 2a by the Project Chemist for 100% of all samples analyzed by the laboratory. Stage 4 will be performed for 10% of the analytical results.

4.3.2. Electronic Data Reporting Formats

Laboratory data will be accepted as a report in PDF format. An Excel electronic deliverable will also be provided.

5. DATA REVIEW, VERIFICATION, AND VALIDATION

Data review is the process by which data are examined and evaluated to varying levels of detail and specificity by a variety of personnel who have different responsibilities within the data management process. It includes verification, validation, and usability assessment. This process ensures the review

activities produce scientifically sound data that are of known and documented quality and meet PQOs used in making environmental decisions.

5.1. Review of Data

All laboratory data packages will include raw data necessary for full validation. Analytical data packages will be validated to Stage 2a by the Project Chemist for 100% of all samples analyzed by the contracted laboratory.

Three distinct evaluative steps will be used to ensure that project-specific data quality needs are met:

- Data Verification (review for completeness) Confirmation by examination and provision of objective evidence that the specified requirements (sampling and analytical) have been completed.
- Data Validation Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Validation is a sampling and analytical process that includes evaluating compliance with method, procedure, or contract requirements and extends to evaluating against criteria based on the quality objectives developed in the QAPP (e.g., the QAPP measurement performance criteria). The purpose of validation is to assess the performance of the sampling and analysis processes to determine the quality of specified data. Data Validation Reports will be generated for each sampling event.
- Data Usability Assessment Determination of the adequacy of data, based on the results of validation
 and verification, and professional judgment by the Project Chemist, for the decisions being made. The
 usability step involves assessing whether the process execution and resulting data meet project quality
 objectives documented in the QAPP.

Data review will be based on laboratory-specific SOPs conforming to the method and applying the principles of the Department of Defense Quality Systems Manual (DoD, 2021) and Data Validation Guidelines (DoD, 2019, 2020a, 2020b), and where applicable and not in conflict, the National Functional Guidelines for Superfund Data Review (USEPA, 2020a-c). If significant deviations arise as a result of initial verification and validation, the level of review will be elevated in order to determine the source and impact of deviations.

5.2. Data Verification and Validation Stages

Data validation and verification stages described below are in accordance with the Department of Defense Data Validation Guidelines (DoD, 2019) and Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA, 2009).

5.2.1. Stage 1

Verification and validation begins with Stage 1 checks of the laboratory analytical data package consisting of compliance of sample receipt conditions, sample characteristics (e.g., percent moisture), and analytical results (with associated information). The following minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 1 validation label:

- (1) Documentation identifies the laboratory receiving and conducting analyses, and includes documentation for all samples submitted by the project or requested for analyses.
- (2) Requested analytical methods were performed and the analysis dates are present.
- (3) Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result (and the uncertainty of each result and clear indication of the type of uncertainty reported if required).
- (4) Requested target analyte result units are reported.
- (5) Requested reporting limits for all samples are present and results at and below the project-specific reporting limits are clearly identified (including sample detection limits if required).
- (6) Sampling dates (including times if needed), date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH and temperature) are documented.
- (7) Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture) to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.2. Stage 2A

Stage 2A validation builds on the validation conducted in Stage 1. Stage 2A validation of the laboratory analytical data package consists of the Stage 1 validation plus the verification and validation checks for the compliance of sample-related QC. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 2A Validation label:

- (8) Requested methods (handling, preparation, cleanup, and analytical) are performed.
- (9) Method dates (including dates, times and duration of analysis for radiation counting measurements and other methods, if needed) for handling (e.g., Toxicity Characteristic Leaching Procedure), preparation, cleanup and analysis are present, as appropriate.
- (10) Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compounds (DMC) recoveries, laboratory control sample (LCS) recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries) are provided and linked to the reported field samples (including the field quality control samples such as trip and equipment blanks).
- (11) Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes) have been added, as appropriate.
- (12) Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.

- (13) Frequency of QC samples is checked for appropriateness (e.g., one LCS per twenty samples in a preparation batch).
- (14) Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.3. Stage 2B

Stage 2B validation builds on the validation conducted in Stage 2A. Stage 2B validation of the laboratory analytical data package consists of the Stage 2A validation plus the verification and validation checks for the compliance of instrument-related QC. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 2B Validation label:

- (15) Initial calibration data (e.g., initial calibration standards, initial calibration verification [ICV] standards, initial calibration blanks [ICBs]) are provided for all requested analytes and linked to field samples reported. For each initial calibration, the calibration type used is present along with the initial calibration equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the initial calibration curve are present, along with their associated percent recoveries, as appropriate (e.g., if required by the project, method, or contract). For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present.
- (16) Appropriate number and concentration of initial calibration standards are present.
- (17) Continuing calibration data (e.g., continuing calibration verification [CCV] standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present.
- (18) Reported samples are bracketed by CCV standards and CCBs standards as appropriate.
- (19) Method specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods).
- (20) Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatographymass spectroscopy [GC-MS] tunes have been run every 12 hours).
- (21) Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

5.2.4. Stage 3

Stage 3 validation builds on the validation conducted in Stage 2B. Stage 3 validation of the laboratory analytical data package consists of the Stage 2B validation plus the recalculation of instrument and sample results from the laboratory instrument responses, and comparison of recalculated results to

laboratory reported results. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 3 Validation label:

- (22) Instrument response data (e.g., GC peak areas) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, matrix spikes, matrix spike duplicates, LCS, and method blanks as well as calibration data and instrument QC checks (e.g., tunes).
- (23) Reported target analyte instrument responses are associated with appropriate internal standard analyte(s) for each (or selected) analyte(s) (for methods using internal standard for calibration).
- (24) Fit and appropriateness of the initial calibration curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the initial calibration curve for each (or selected) analyte(s) from the instrument response.
- (25) Comparison of instrument response to the minimum response requirements for each (or selected) analyte(s).
- (26) Recalculation of each (or selected) opening and closing CCV (and CCB) response from the peak data reported for each (or selected) analyte(s) from the instrument response, as appropriate.
- (27) Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated initial calibration response for each (or selected) analyte(s).
- (28) Recalculation of percent ratios for each (or selected) tune from the instrument response, as appropriate.
- (29) Compliance check of recalculated percent ratio for each (or selected) tune from the instrument response.
- (30) Recalculation of each (or selected) instrument performance check (e.g., instrument blanks,) from the instrument response.
- (31) Recalculation and compliance check of retention time windows (for chromatographic methods) for each (or selected) analyte(s) from the laboratory reported retention times.
- (32) Recalculation of reported results for each reported (or selected) target analyte(s) from the instrument response.
- (33) Recalculation of each (or selected) reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries) from the instrument response.
- (34) Each (or selected) sample result(s) and spike recovery(ies) are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.

Note: Selection of analytes, spikes, and performance evaluation checks for the Stage 3 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including (but not limited to) the type of verification and validation being performed (manual or electronic), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

5.2.5. Stage 4

Stage 4 validation builds on the validation conducted in Stage 3. Stage 4 validation of the laboratory analytical data package consists of the Stage 3 validation plus the evaluation of instrument outputs. The following additional minimum baseline checks (as relevant) shall be performed on the laboratory analytical data package received for a Stage 4 Validation label:

- (35) All required instrument outputs (e.g., chromatograms, mass spectra) for evaluating sample and instrument performance are present.
- (36) Sample results are evaluated by checking each (or selected) instrument output (e.g., chromatograms, mass spectra) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences).
- (37) Each (or selected) instrument's output(s) is evaluated for confirmation of non-detected or tentatively identified analytes.

Selection of instrument outputs for the Stage 4 validation checks for a laboratory analytical data package being verified and validated generally will depend on many factors including, but not limited to, the type of verification and validation being performed (electronic or manual), requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract, the number of laboratories reporting the data, the number and type of analytical methods reported, the number of analytes reported in each method, and the number of detected analytes.

5.3. Data Verification and Validation Stages

A data validation report will be generated by the USACE Chemist that encompasses the results of the manual review of private lab data. The data validation report will be an appendix to the Final Report. Professional judgment shall be used when deciding if qualification of data is applicable. When professional judgment is applied, the rationale shall be provided. Tables of qualified data and the reasons for qualification will also be included in the data validation report.

Qualifiers will be added to data during the review as necessary. Qualifiers applied to the data as a result of the review are as follows:

U Indicates the compound or analyte was analyzed for but not detected at or above the stated limit. The data are usable for decision-making purposes.

- UJ Indicates the compound or analyte was analyzed for but not detected. Due to a quality control deficiency identified during data validation, the value reported may not accurately reflect the sample quantitation limit. The associated value is considered estimated, but the data are generally usable for decision-making purposes.
- J Indicates the compound or analyte was analyzed for and detected. The associated value is estimated due to a quality control deficiency identified during data validation. False positives or false negatives are unlikely to have been reported and the data are generally usable for decision-making purposes.
- J+ Data are qualified as estimated with a high bias. False positives are likely to occur but the data are generally usable for decision-making purposes.
- J- Data are qualified as estimated with a low bias. False negatives are likely to occur but the data are generally usable for decision-making purposes.
- X The sample results (including non-detects) were affected by serious deficiencies in the ability to analyze the sample and to meet published method and project quality control criteria. The presence or absence of the analyte cannot be substantiated by the data provided. Acceptance or rejection of the data should be decided by the project team (which should include a project chemist), but exclusion of the data is recommended.
- R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified. Rejection of the data should be decided by the project team (which should include a project chemist).

Note 1: It is possible that J-qualified data are not suitable for some purposes. For example, a J-qualified concentration with a low bias that is just below a screening value may not be usable to determine whether the analyte concentration is above or below the screening value. The effect of the use of qualified data on the decision-making process must be evaluated as part of the "reconciliation with user requirements" process.

Note 2: Estimated maximum possible concentration (EMPC-qualified) values will be treated as detected concentrations and flagged by the laboratory. EMPC-qualification is used when mass spectrometry results meet all of the identification criteria in the method except the ion abundance ratio criteria. The results will be flagged J+ by the validator, to indicate that the reported concentration is detected and may be associated with a possible bias.

5.4. Usability Assessment

The Project Chemist will evaluate overall precision, accuracy, completeness, representativeness, comparability, and sensitivity of the sampling data; including an assessment of the overall usability of the data and describing any limitations on its use. The Project Chemist will summarize any audit information,

indicating corrective actions taken. This information will be part of the data validation report, which is an appendix to the Final Report.

5.4.1. Precision

Precision is defined as the degree of agreement between or among independent, similar, or repeated measures. Duplicate pairs such as MS/MSD, LCS/LCSD, laboratory duplicate, and field duplicate samples are evaluated as RPD and are compared to the limits of the DoD QSM, if present, and then to the laboratory's method limits. The relative percent difference (RPD) for these analyses is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{S_{avg}} \times 100\%$$

Where S_1 and S_2 = the observed concentration of analyte in the sample and its duplicate, and

 S_{avg} = the average of observed analyte concentration in the samples and its duplicate.

5.4.2. Accuracy

Accuracy is the amount of agreement between a measured value and the true value. Accuracy, expressed as %Recovery (%R), is assessed for each method, analyte, and matrix, by comparing MS, MSD, LCS, LCSD, and surrogate recoveries to the method limits.

5.4.3. Representativeness

Representativeness is a qualitative parameter that expresses the degree to which the sample data are characteristic of a population. Blank samples identify compounds that may have been introduced into the samples during preparation, or analysis. Representativeness is addressed by evaluating blank samples, sample custody, and holding times and temperatures.

5.4.4. Completeness

Analytical completeness is expressed as the percentage of measurements that were judged to be valid, i.e., not rejected, and acceptable for all intended date use. <u>The analytical completeness goal for this sampling event is 95%.</u>

5.4.5. Sensitivity

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. The sensitivity of the analytical methods (i.e., method reporting limits) identified for this project are evaluated against the QAPP.

6. REFERENCES

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